June 7, 1960 G. B. LUNDEVALL 2,939,783

ZINC REFINING

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Fig. 1.

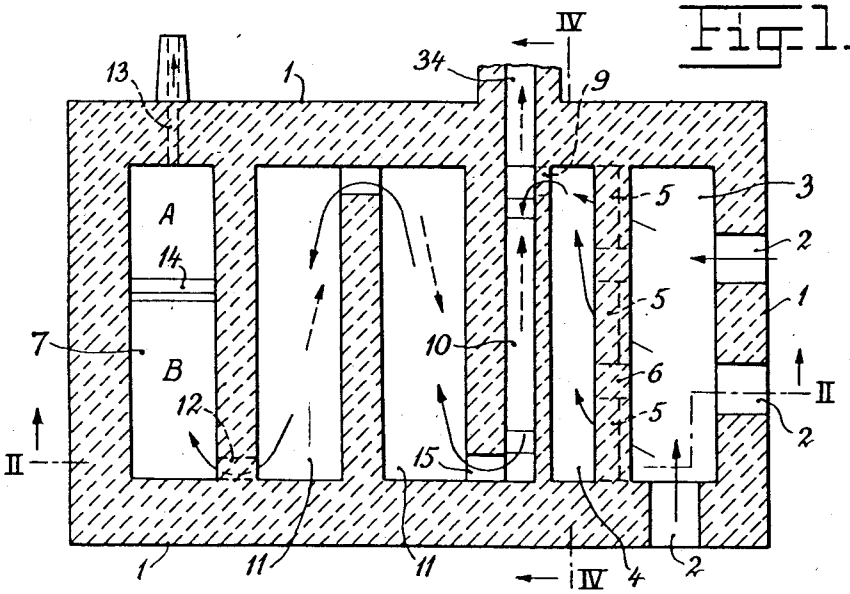
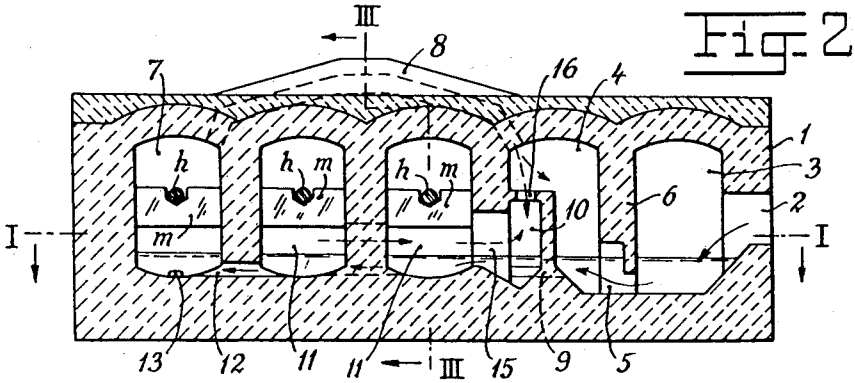


Fig. 2.



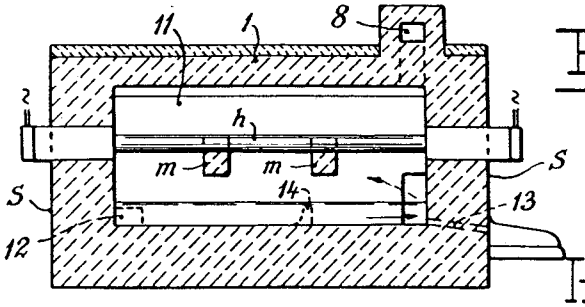


Fig. 3.

Inventor
Gustav Blom Lundevall
Hill Sherman Meroni Gross & Simpson
by Attys

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Fig. 4.

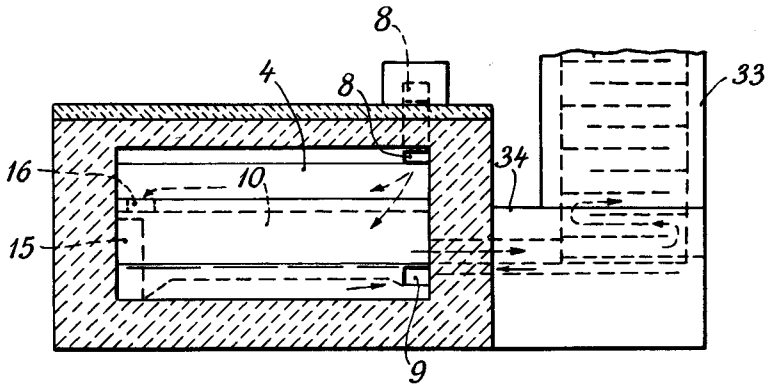
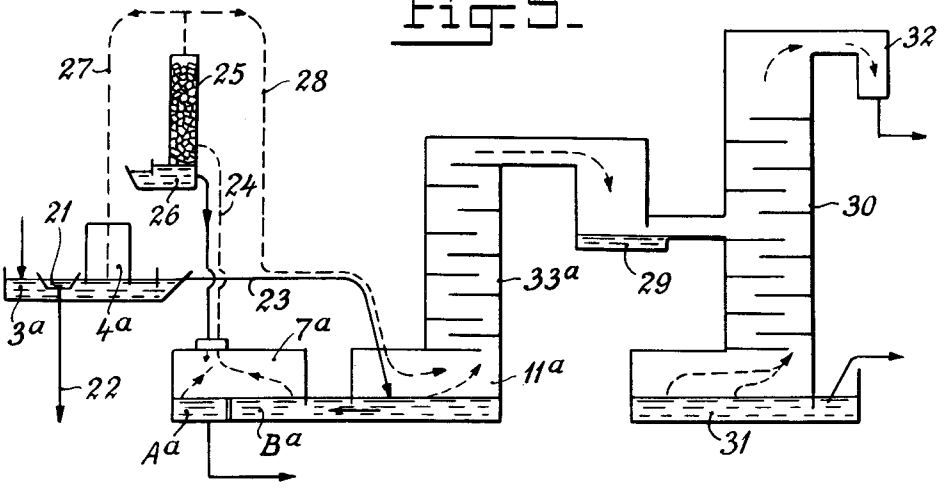


Fig. 5.



Inventor
Gustav Blom Lundevall
by Hill Sherman Meroni Gross & Simpson
Attys

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ZINC REFINING

Gustav Blom Lundevall, Hoffsgate 17, Larvik, Norway

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Claims priority, application Norway May 22, 1957

17 Claims. (Cl. 75—88)

This invention relates to a process and apparatus for refining impure zinc and old die cast alloys. Its object is to provide a continuous process and apparatus for zinc refining affording substantial advantages with respect to heat economy and rendering a zinc metal of high purity and in good yield as well.

In a preferred mode of carrying out the process according to the invention, the zinc-bearing metal to be refined is melted in a first processing step and conducted to a first distillation zone from where nearly pure zince vapors are obtained, and from where the remaining molten metal is conducted to a second distillation zone, where heat is applied sufficient to vaporize substantially all or at least a major part of the zinc contained in the melt, these vapors being returned to the first processing step and the impure molten metal residue being drained from the second distillation zone.

The vapors from the second distillation zone, if highly contaminated, may be subjected to a fractional condensation to remove high boiling point impurities before the vapors are returned to the first processing step. These vapors, or a part thereof, are advantageously condensed on liquid metal in the first processing step, whereby the latent heat of the vapors is utilized for melting the charge which, together with the condensate, flows into the first distillation zone.

In this distillation zone a selective evaporation takes place from the molten metal flowing through it, which, together with the increase in zinc concentration resulting from said condensation of zinc vapors coming from the second distillation zone, produces zinc vapors of a high purity, which vapors, on leaving the zone and if desired, may easily be purified to give a special high-grade zinc of 99.99+ percent purity.

By applying the well known principle that iron and aluminum together form the compound Al_3Fe, which, owing to its low density, accumulates on top of the zinc melt, a preliminary purification of the melt may be effected, by which harmful iron is removed. If not removed in advance, such iron, which has a low solubility in boiling zinc, might cause serious difficulties during the following extraction of the zinc by a distillation process, the iron having a high melting point and being difficult to remove.

The process according to the invention will now be explained in detail in connection with the drawings, which diagrammatically illustrate embodiments of the invention.

Fig. 1 is a horizontal section of a furnace adapted for application with one embodiment of the invention.

Fig. 2 is a vertical section taken along the line II—II in Fig. 1.

Fig. 3 is a vertical section taken along the line III—III in Fig. 1.

Fig. 4 is a vertical section taken along the line IV—IV in Fig. 1.

Fig. 5 is a diagrammatic drawing serving to explain the principles of the invention and illustrating a further embodiment including some additions and modifications with respect to that illustrated in Figures 1 to 4.

Solid arrows in the drawings indicate directions of solids or liquid flow, while dotted arrows indicate vapor flow.

In Figures 1 to 4 the furnace, which is made of a fireproof material, is designated by **1**. The metal to be refined, advantageously consisting of raw zinc, old zinc or die cast scrap is fed into the melting chamber **3** through the openings **2**. The liquids in the melting chamber **3** and the condensation chamber **4** communicate through submerged openings **5** in the lower part of the partition wall **6**.

Zinc vapors from the distillation furnace **7**, which will be described later, have access to the condensation chamber **4**, which is inaccessible from the outside, through the duct **8** and are condensed on the molten metal, the latter thereby receiving the latent heat of condensation of the vapors, whereby, owing to the circulation and heat conductivity of the melt, the metal charged in the chamber **3** can be melted.

A zinc pump of adjustable velocity may, if desired, easily be installed at the exit of the melting chamber **3** in order to circulate the molten metal between the chambers **3** and **4** and thereby achieve a suitable heat transfer.

The metal charged is subjected to a preliminary purification in the melting chamber **3**, from which impurities in the form of ash, pieces of iron and the like easily can be raked out, or, if the supplied impure metal contains both iron and aluminum, crystals of said Al_3Fe may be formed at suitable temperatures, constituting a layer on top of the melt, and may be removed in the form of a mush.

The partition wall **6**, as indicated in Fig. 2, extends into the melt to a depth of about 15 cm. (6 inches). This prevents said impurities from finding their way into the condensation chamber **4**, from which the pre-purified metal by displacement is caused to flow through the lower passage **9** and via a duct **10** to the first distillation furnace **11**. When slowly flowing through the duct **10** the metal is heated to a temperature near the boiling point of zinc (906 centigrade) by the countercurrent flow of superheated vapors leaving the distillation furnace **11**.

The heat required by the distillation furnaces **11** and **7** may be supplied in various known ways, for example by the combustion of gas or oil in a chamber above the melt, but separated from the latter by a curved or arched ceiling made from SiC-stone, which provides for a very satisfactory heat conductivity and radiation to the metal bath beneath. In the preferred embodiment shown the heating is effected by means of graphite resistors *h*, which have been employed by the inventor for years and with good results. These are mounted on supporting walls and arches *s* and *m* respectively in the furnace chambers where they are protected by the zinc atmosphere. A uniform heat of radiation, simple means of regulation and a stable performance are thereby achieved.

The distillation furnace **11** may consist of one chamber or several interconnected chambers (Figures 1 and 2 showing two chambers as an example). Due to the high zinc content of the melt in furnace **11** the temperature will adjust itself to that of boiling zinc (906 centigrade), whereby pure zinc vapors are evolved and permitted to escape through the opening **15** and the duct **10**, from where the vapors may enter a lead fractionating column **33** before being condensed.

The impurities having higher boiling points than zinc will remain in the melt of furnace **11** and become more concentrated towards the exit.

It is a significant feature of this invention, however, that a relatively high zinc content is maintained in the

melt leaving furnace **11**, via the submerged passage **12**, so that zinc vapors of a high purity can be produced.

The second distillation zone, i.e. furnace **7**, has the purpose of extracting excess zinc from the melt entering from furnace **11** and to concentrate the impurities in the molten residue, which may be removed without an appreciable loss of zinc.

This requires a higher temperature than found in furnace **11**, and consequently the vapors driven off from furnace **7** will be relatively impure, which is not of prime significance since they are to be condensed and redistilled as already mentioned. In order to prevent these vapors from mixing with the pure vapors from the furnace **11** and thereby contaminating the product, it is an important feature of the invention that substantially all of the impure vapors are condensed, and further that the heat required for melting the charge as well as heat required to cover losses from the melting and condensation chambers, is supplied by condensation of zinc vapors coming from the second distillation furnace **7**, all of which results in that the melt flowing into the first distillation furnace **11** consists of a mixture of pre-purified molten metal plus an even purer condensate. This melt will, therefore, contain a higher percentage of zinc than the metal charged and, moreover, the rate of flow of molten metal into the first distillation furnace **11** will be higher than the feed rate of the charge. These advantages are achieved without sacrificing any part of the heat economy.

The metal residue in the second distillation furnace **7** is discharged through the outlet **13**. In order to facilitate this operation, which is not necessarily continuous, the second distillation furnace **7** may be divided by a weir **14** so as to form two basins A and B. The hot melt overflowing the weir **14** will contain a high percentage of impurities and correspondingly less zinc, practically all of which is driven off during the time when the melt remains in basin A, where the temperature is relatively high and adjusted to give satisfactory zinc extraction, preferably by control of the electric power input. The liquid level in the basin A far from the inlet **12** may be lower than that of the basin B, so that if the residue is discharged intermittently from basin A through outlet **13**, the deeper melt in the basin B will still constantly contain sufficient zinc to maintain a considerable vapor flow through the duct **8**. This tapping operation requires a relatively short period of time, and by the method described the distillation process going on continuously in the furnace **11** need not be disturbed.

The following calculation gives information about the weight ratio of metal charged and condensing zinc vapors supplying the necessary heat to the melt inclusive of losses. As an example an hourly charge of 556 kgms. of remelted die cast scrap containing about 91.5% Zn is assumed.

	Cal./h.
For melting and heating to about 650 centigrade about 108 cal./kg. are required, or	About 60,000
To which are added heat losses estimated to	About 60,000
Heat required	About 120,000

In order to supply 120,000 cal./h. about 277 kgms. of zinc vapor must be condensed. In the example given, the charge amounts to 556 kgms. of metal alloy per hour. Adding the 277 kgms. of zinc condensate it follows that 833 kgms. of metal per hour will flow from chamber **4** to the distillation furnace **11**.

In practice the evaporation of zinc in the distillation furnace **7** can only approximately be adapted to the amount of zinc condensing in the chamber **4**. However, an exact adjustment in this respect is not required, since the invention permits zinc vapors from the first distillation furnace **11** to flow into the duct **10** through the opening **15** and further through the opening **16** (Fig. 4) into the condensation chamber **4** if the pressure in the latter chamber should be reduced by too slow evaporation in the distillation furnace **7**. Inversely, should excess vapors from the furnace **7** find their way into the duct **10**, no harm would be done. This comes from the fact that the relatively impure vapor from the second distillation furnace **7** is purified in passing through the condensation chamber **4**, contaminating metals with a higher boiling point than zinc precipitating owing to the favorable conditions provided in this chamber for a fractional condensation. This is considered to be an important feature of the present invention.

Referring to the example above, an approximate material balance would appear as follows (charge weight after removal of dross and mush):

	Total Kgms./h.	Zinc		Impurities	
		Kgms.	Percent	Kgms.	Percent
Charge supplied	556	506	91.5	50	9.0
Zinc condensate returned from the second distillation furnace 7	277	277	about 100		
Melt entering the first distillation furnace 11	833	783	94	50	6
Product or zinc vapors expelled	500	500	100		
Melt entering the second distillation furnace 7	333	283	85	50	15
Zinc vapors returned to the condensation chamber 4	277	277	about 100		
Metallic residue discharged from the second distillation furnace 7	56	6	10.7	50	89.3
Estimated yield of zinc	500/506.100 equalling 99%				

Note that in the first distillation furnace **11** the melt will on an average contain

$$\frac{94+85}{2}$$

about 89.5% zinc. This percentage is so high that the zinc vapor will be of a relatively very high purity, which will facilitate a possible subsequent refining in fractionation columns with the purpose of removing impurities such as lead and cadmium, so as to produce a metal containing 99.99+ percent Zn.

In Fig. 4 is shown how a "lead fractionating column" may be adapted to the apparatus of the invention, with a view to such further purification. Zinc vapors from the first distillation furnace **11** enter the column **33** via the opening **15** and the duct **10**, giving off excess heat along this passage.

Using graphite heating elements, the energy consumption in refining 500 kgms./h. of zinc, including heat losses from the furnaces as well as from a subsequent "lead column," is, roughly:

1st distillation furnace 11:		
(a) Vaporization of	500 kgms. zinc	
(b) Vaporization of	200 kgms. zinc reflux for "the lead column"	
	700 kgms. zinc	370 kw.
Transformer and furnace losses estimated to		About 110 kw.
Sum		About 480 kw.
2nd distillation furnace 7:		
(c) Vaporization of 277 kgms. zinc		150 kw.
Transformer and furnace losses estimated to		About 90 kw.
Sum		About 240 kw.
Energy requirement for both furnaces: Total		About 720 kw.

which, with a production of about 500 kgms. of refined zinc per hour, corresponds to about 1440 kwh./1000 kgms. zinc.

The embodiment shown diagrammatically in Fig. 5 largely comprises the same elements as have been described above, with certain modifications and additions, and corresponding elements have been given the same designation numerals with the addition *a*, and may be constructed similarly and from the same materials as indicated in connection with Figures 1 to 4.

In the embodiment shown in Fig. 5 the metal to be refined is fed into the melting chamber 3*a*, which is in liquid communication with the condensation chamber 4*a* so that the metal in the chamber 3*a* may be melted when the chamber 4*a* is heated. In the melting chamber 3*a* impurities forming a mush 21 on top of the melt are skimmed off and removed through a channel 22. The mush may then be cast into pig metal for separate treatment or subjected to distillation for instance by being conducted still hot to basin A*a* of the distillation furnace 7*a*.

The pre-purified melt coming from the condensation chamber 4*a* is conducted through a channel 23 and into the distillation furnace 11*a* in which purified zinc vapors are evolved and conducted through the column 33*a*. This column serves to isolate metals the boiling points of which are higher than 906 centigrade and is mounted for cooperation with the first distillation furnace 11*a*, so that the condensate from the column 33*a* is returned to the furnace 11*a*. A correspondingly higher amount of zinc vapor must then be distilled from the furnace 11*a* and so that the latent heat of condensation will cover the loss of heat from the column and maintain a reflux required for fractionation.

The zinc vapors from "the lead column" 33*a* are condensed in a condenser 29 and fed into a column 30 in any known manner. The column 30 is a fractionation column mounted on top of a separate distillation furnace 31 and serves to remove impurities with lower boiling points than zinc, for instance cadmium. Such impurities are condensed in the condenser 32, while zinc of a purity of 99.99+ percent is tapped from the furnace 31 at the foot of the column 30. If desired, the purified zinc may then flow into an alloy sump from where primary die cast metal is cast into ingots.

The distillation furnace 11*a* is heated in the same way as described in respect of the furnace 11. The remaining melt is continuously conducted to the distillation furnace 7*a*, which is heated in the same way as the furnace 7 previously described.

The zinc vapors evolved in the furnace 7*a* are conducted through the channel 24, and if they contain a relatively large amount of higher boiling impurities, the vapors may advantageously be conducted through a fractionation column 25, in which such impurities (for instance lead) are condensed and return from the foot 26 of the column 25 to the furnace 7*a*. A part of the vapors thus pre-purified are conducted through the channel 27 and into the condensation chamber 4*a* in which the vapors are condensed on molten metal which is thereby heated to a higher temperature owing to the latent heat of condensation being absorbed. As already mentioned this heat is utilized for melting the metal to be refined. The part of the zinc vapors coming from the furnace 7*a* and being purified in the column 25 and which are not required for supplying heat to the chamber 4*a*, are returned to the distillation furnace 11*a* vie the channel 28. Vapors and condensate may be conducted into the furnace 11*a* together with the main stream of molten metal being supplied through the channel 23, whereby the temperature of the melt is raised owing to the condensation of zinc vapors.

Should the supply of zinc vapors from the furnace 7*a* be insufficient for providing the heat required for the melting process in the condensation chamber 4*a* and the melting chamber 3*a*, then zinc vapors may also be conducted thereto from the furnace 11*a* via the channels 27 and 28.

Owing to the selectivity of the distillation taking place in the furnace 11*a* and the fractionating effect of the lead column, the zinc vapors from the column 33*a* will be of a high purity and may, if desired, be processed further in the column 30 for removal of impurities with lower boiling points than zinc. A zinc of particularly high purity, e.g. 99.99+ percent, may thus be obtained.

Although the embodiments of Fig. 5 comprise various features not shown for the furnace structure of Figs. 1 to 4, it will be understood that any or all such additions may be combined with this structure. Especially, the vapor outlet 34 in Fig. 2 may be connected to a reflux column such as 33*a*, followed, if desired, by a "cadminum column" such as 30.

I claim:

1. A continuous process of refining impure zinc which comprises melting a charge of zinc-bearing alloy, conducting the resultant melt to a first distillation zone, maintaining the temperature of the melt in said zone substantially at the boiling point of the zinc thereby generating purified zinc vapors, withdrawing said vapors from said zone, continuously conducting melt from said zone further to a second distillation zone, maintaining the temperature of the latter sufficiently high to vaporize nearly all of the remaining zinc thereby generating impure zinc vapors of a higher temperature than that of said purified vapors, condensing vapors produced in said second zone, continuously returning the resultant condensate to said first distillation zone therein subjecting said condensate to redistillation together with the molten charge, and withdrawing impure molten metal residue from said second distillation zone.

2. A process according to claim 1, in which the condensation of impure vapors generated in said second distillation zone is effected on the melt passing to the first distillation zone.

3. A process according to claim 1, in which a layer of impure mush forming on top of the freshly molten charge is removed therefrom before the resulting melt is contacted with vapors from the second distillation zone.

4. A continuous process of refining impure zinc which comprises melting a charge of zinc-bearing alloy, conducting the resultant melt to a first distillation zone, maintaining the temperature of the melt in said zone substantially at the boiling point of the zinc thereby generating purified zinc vapors, withdrawing said vapors from said zone, continuously conducting melt from said zone further to a second distillation zone, maintaining the temperature of the latter sufficiently high to vaporize nearly all of the remaining zinc thereby generating impure zinc vapors of a higher temperature than that of said purified vapors, condensing vapors produced in said second zone, continuously returning the resultant condensate to said first distillation zone, whereby the overall purity and weight of the liquid zinc supplied to the first distillation zone is made to exceed the purity and weight of the incoming charge, subjecting the said condensate to redistillation together with the molten charge, and withdrawing impure molten metal residue from said second distillation zone.

5. A continuous process of refining impure zinc which comprises melting a charge of zinc-bearing alloy in a melting zone, conducting the resultant melt via a conditioning zone to a first distillation zone, maintaining liquid communication and preventing gaseous communication between said melting zone and said conditioning zone, maintaining the temperature of the melt in said distillation zone substantially at the boiling point of zinc thereby generating purified zinc vapors, withdrawing said vapors from said zone, continuously conducting melt from said zone further to a second distillation zone, maintaining the temperature of the latter sufficiently high to vaporize nearly all of the remaining zinc thereby generating impure zinc vapors of a higher temperature than that of said purified vapors, condensing vapors produced in said second zone, continuously returning the resultant condensate to said first distillation zone, therein subjecting said condensate to redistillation together with the molten

charge, and withdrawing impure molten metal residue from said second distillation zone.

6. A process according to claim 5, in which a part of the liquid metal flowing from the melting zone to the conditioning zone is forced to return to the former.

7. A continuous process of refining impure zinc which comprises melting a charge of zinc-bearing alloy in a melting zone, conducting the resultant melt via a conditioning zone to a first distillation zone, maintaining liquid communication and preventing gaseous communication between said melting zone and said conditioning zone, maintaining the temperature of the melt in said distillation zone substantially at the boiling point of the zinc thereby generating purified zinc vapors, withdrawing said vapors from said zone, continuously conducting melt from said zone further to a second distillation zone, maintaining the temperature of the latter sufficiently high to vaporize nearly all of the remaining zinc thereby generating impure zinc vapors of a higher temperature than that of said purified vapors, condensing vapors produced in said second zone on the melt in said conditioning zone, thus continuously returning the resultant condensate to said first distillation zone and subjecting it to redistillation together with the molten charge, maintaining the amount of condensing vapors sufficiently high relative to the weight of the incoming charge to melt the latter and heat the same to a temperature near the boiling point of the zinc by means of the heat of condensation, and withdrawing impure molten metal residue from said second distillation zone.

8. A process according to claim 7, in which the conditioning zone is maintained in gaseous communication with the entrance to the first distillation zone.

9. A process according to claim 7, in which the vapors evolved in the second distillation zone are purified by a preliminary fractional condensation before being allowed to condense in the conditioning zone.

10. A process according to claim 9, in which zinc vapors having been purified by a preliminary fractional condensation are mixed with the zinc vapors generated in the first distillation zone.

11. A zinc refining apparatus comprising, in combination, means forming a substantially closed chamber for melting a continuous charge of impure zinc, means forming a contiguous second chamber having means for entering metal vapors thereto, means forming submerged passages between said chambers for molten metal to circulate between them means forming a third chamber for distilling zinc from a molten alloy, means for continuously conducting such molten alloy from said second to said third chamber, means associated with the latter for heating the melt therein to substantially the boiling temperature of the zinc, means for removing the resultant vapors of purified zinc, means forming a fourth chamber, means for continuously conducting molten alloy from said third chamber to said fourth chamber, means associated with the latter for heating the melt therein sufficiently to distil off nearly all of its zinc content, means for conducting at least a considerable part of the resultant impure zinc vapors from said fourth chamber to said second chamber,

and means for discharging molten metal residue from said fourth chamber.

12. Apparatus according to claim 11, further comprising mechanical means for circulating said molten metal between said melting chamber and said second chamber.

13. Apparatus according to claim 11, in which said third and fourth chambers are provided with electrical heating means.

14. Apparatus according to claim 13, in which said heating means comprises a resistance element of substantially graphite or carbon material.

15. A zinc refining apparatus comprising, in combination, means forming a substantially closed chamber for melting a continuous charge of impure zinc, means forming a contiguous second chamber having means for entering metal vapors thereto, means forming submerged passages between said chambers for molten metal to circulate between them, means forming a third chamber for distilling zinc from a molten alloy, means for continuously conducting such molten alloy from said second to said third chamber, means associated with the latter for heating the melt therein to substantially the boiling temperature of the zinc, means for removing the resultant vapors of purified zinc, means forming a fourth chamber, means for continuously conducting molten alloy from said third to said fourth chamber, means associated with the latter for heating the melt therein sufficiently to distil off nearly all of its zinc content, means for discharging molten metal residue from said fourth chamber, condensing means for fractionally condensing impure zinc vapors evolved in said fourth chamber, means for withdrawing the resultant condensate from said condensing means, and means for conveying remaining zinc vapors from said condensing means to said second chamber.

16. A zinc refining apparatus, comprising, in combination, means forming a first distillation chamber, means for introducing molten metal into said chamber, means associated with said chamber for heating the melt therein so as to distill off a substantial part of its zinc content, means for withdrawing vapors thus evolved, a second distillation chamber, means for continuously conveying remaining melt from said first to said second chamber, means associated with said second distillation chamber for heating the melt therein sufficiently to distill off nearly all of its zinc content, means for conveying zinc vapors thus evolved into contact with melt passing to said first distillation chamber, and means for withdrawing impure liquid residue from said second distillation chamber.

17. An apparatus according to claim 16, in which a weir is provided in said second distillation chamber between the outlet of said melt conveying means and the inlet of said means for removing liquid residue.

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